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A FUNDAMENTAL STUDY OF P/M (POWDER METALLURGY)
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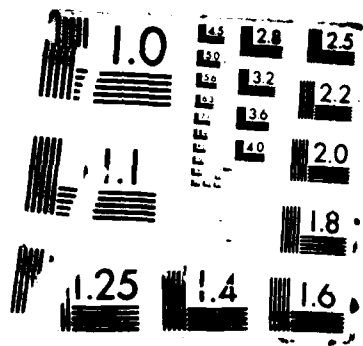
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ANNUAL TECHNICAL REPORT

"A FUNDAMENTAL STUDY OF P/M PROCESSED
ELEVATED TEMPERATURE ALUMINUM ALLOYS"

AFOSR GRANT #82-0010

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Microstructural stability is a function of the powder processing temperatures for degassing and extrusion. In addition, the future plans concerning Al-Fe-Ce alloys are discussed in relation to the current program.

ABSTRACT OF RESULTS

Al-Fe-Ni powder containing 0.19 volume fraction of FeNiAl₃ dispersoid (~0.18 μm) was mechanically alloyed (MA) and hot extruded. There is a significant enhancement in creep response compared to the same alloy without MA and microstructural stability is superior. Improvements in creep strength and microstructural stability are attributed to the presence of fine-scale (~30 nm) oxides/carbides introduced during MA distributed uniformly throughout the matrix, at matrix- intermetallic interfaces, and on subgrain boundaries. The fine-scale dispersion inhibits grain and intermetallic coarsening and appears to be related with the diffusing Fe and Ni atoms. Microstructural stability is a function of the powder processing temperatures for degassing and extrusion. Future efforts are directed to the understanding of microstructural stability and studies of mechanically alloyed Al-Fe-Ce.

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I. THE PRESENT PROGRAM

In aerospace structural applications, there is a need for high performance aluminum alloys with improved properties and elevated temperature stability. A goal of the Air Force is to develop aluminum alloys for long-time service in the temperature range 230-340°C. There are severe inherent limitations to these property and performance goals utilizing ingot metallurgy (I/M) and this has stimulated research into powder metallurgy (P/M) as a processing alternative. Of particular promise and potential are the P/M technologies of rapid solidification (RS) and mechanical alloying (MA). It has been clearly demonstrated that P/M processing provides enhanced alloying flexibility and results in fine-scale homogeneous microstructures with minimal attendant solute segregation.

The key to improved high temperature strength is the presence of a fine-scale thermodynamically stable dispersion, uniformly distributed in the alloy matrix. Transition metal elements are added to aluminum to produce dispersions of stable, hard intermetallics which are resistant to coarsening. Specifically, Co or Fe and Ni give rise to dispersoids of Co_2Al_9 or FeNiAl_9 (10-12). The degree of homogeneity, size, and volume fraction of the dispersoid are limited in I/M alloys by virtue of the inherent low rates of solidification. P/M processing involves rapid solidification of the air atomized melt which ensures a uniform, fine-scale dispersoid in the aluminum matrix. This results in a significant increase in strength by virtue of (i) extending solid solubility and thereby increasing the volume fraction of dispersoid, and (ii) a fine stabilized grain structure of grain size $\sim 1\mu\text{m}$.

In the initial phase of this program, we have established optimum conditions for the P/M processing, resulting microstructures and mechanical properties of prealloyed Al-Fe-Ni. Three volume fractions of FeNiAl_9 dispersoid were studied, namely 0.19, 0.25 and 0.32. ambient and elevated temperature tensile and creep response were assessed at temperatures up to 400°C and microstructural stability was evaluated at

temperatures up to 500°C.

In order to further enhance the properties and performance of this new generation of P/M aluminum alloys, rapidly solidified powders have been subjected to mechanical alloying (MA) prior to hot consolidation. MA was developed at INCO in order to prepare oxide dispersion strengthened superalloys. This progress report summarizes studies to-date in which the Al-Fe-Ni prealloyed powder was mechanically alloyed prior to consolidation. Elevated temperature creep response hardness, and microstructural stability have been examined up to 500°C.

II. PROGRAM SUMMARY

On the basis of completed efforts this program on powder processed Al-Fe-Ni alloys, three areas are identified in which more basic research is needed. These are: (i) an understanding of operative creep deformation modes/mechanisms in the temperature range 200-300°C, (ii) the origin of the improved strength and creep resistance of the mechanically alloyed material, e.g. Al-Fe-Ni, Al-Fe-Ce, (iii) the origin of the enhancement in microstructural stability as a result of mechanical alloying. Each area is considered critical, if the promise and utility of powder processed wrought dispersion strengthened Al-base alloys are to be fully exploited. The status of the current on-going program is reviewed and proposed future studies on Al-Fe-Ce is rationalized.

III. INTRODUCTION

Improvements in ingot metallurgy (I/M) aluminum alloys in terms of high temperature strength have been hampered by microstructural limitations. The modern aerospace industry mandates high performance materials with increased high temperature structural stability. The powder metallurgy (P/M) approach is an attractive alternative; intrinsic benefits include increased alloying flexibility, microstructural homogeneity, and minimal solute segregation, compared to I/M alloys (1,2).

A thermodynamically stable second phase precipitated in aluminum can improve strength. This effect can be further enhanced if the second phase, in the form of a dispersoid, is distributed uniformly throughout the matrix. The homogeneity, size and volume fraction of the dispersoid are limited in conventionally cast aluminum alloys. P/M processing involves rapid solidification of the air atomized melt. Thus, it is possible by this means to achieve a high degree of homogeneity, size uniformity, and fine scale of the dispersoid (3,4). Dispersions of hard, thermodynamically stable intermetallics have been found to precipitate in aluminum alloys containing cobalt, or iron and nickel (5-9); examples are Co_2Al_9 and $(\text{FeNi})\text{Al}_9$. Powder processing of these alloys can yield not only a fine stabilized grain size ($1\mu\text{m}$), but also an increase in the volume fraction of the second phase particles through extension of solid solubility limits.

P/M processing of rapidly solidified alloys in the Al-Fe-Ni system has been studied. In these alloys, the $(\text{FeNi})\text{Al}_9$ dispersoid was present in volume fractions 0.19 to 0.32 (10,11). Tensile and creep response were evaluated at temperatures up to 400 C, and the corresponding microstructures and microstructural stability were assessed at temperatures as high as 500 C.

A fine scale uniform distribution of dispersoids can also be introduced into a metal/alloy powder by mechanical alloying (MA). This P/M processing technique developed by Benjamin (12,13) has been applied to aluminum (14-17), and aluminum alloys (18,19), and has been shown to enhance strength and strength retention at elevated temperatures. Dispersoids in the aluminum and aluminum alloys were identified as Al_2O_3 and Al_4C_3 (17-19). The source of carbon is known to be the process-control agent added prior to MA to prevent excessive welding (17).

Recently, the authors have characterized the effect of MA on P/M processed Al-Fe-Ni with respect to strength and microstructural stability (20). Processing parameters (degassing and extrusion temperature) were varied, but the volume fraction of $(\text{FeNi})\text{Al}_9$ was kept constant at 0.19. Considerable improvement in strength

was apparent, compared to the non-MA material (10,11). Furthermore, exposing the MA alloys at 450 C for as long as 624 hours showed no significant change in strength or microstructure (20).

The purpose of the study reported here has been to characterize the creep response of MA Al-Fe-Ni; the material was identical to that used in the study cited previously (20). In addition, microstructural stability was assessed at temperatures above 500 C.

IV. EXPERIMENTAL PROCEDURES

(a) Material Processing and Characterization

Briefly, Al-Fe-Ni powder was air atomized, mechanically alloyed, canned, degassed, and hot extruded to full density; details are given elsewhere (20). The MA powders were processed using three combinations of outgassing temperature and extrusion temperature in the range 370 C to 490 C, at a constant extrusion ratio of 16:1. The three combinations are designated : **a** (low degassing temperature, high extrusion temperature), **b** (low degassing temperature, low extrusion temperature), and **c** (high degassing temperature, low extrusion temperature). Microstructure and properties were examined in the as-extruded condition and after elevated temperature exposure at 550 C (up to 288 hours) and 610 C (50 hours), via optical microscopy and transmission electron microscopy (TEM). Details of the preparation procedures are given elsewhere (20).

(b) Mechanical Testing

Hardness was determined at ambient temperature in the as-extruded condition, and following elevated temperature exposure. Creep tests were performed in air under a constant tensile load using a lever arm creep testing machine. Threaded end specimens with a 32 mm gage length and a 6.35 mm gage diameter were used with specimens tested parallel to the direction of extrusion. Creep strains were monitored

with an LVDT that measured the displacement of the grip linkage outside the furnace. Rupture life was determined as a function of stress and temperature. Creep tests that ran in excess of 200 hours were terminated.

V. RESULTS

(a) Microstructure

Representative micrographs showing the as-extruded microstructure of the MA Al-Fe-Ni material are shown in Figure 1. The fine oxide/carbide dispersions are seen to be uniformly distributed in the aluminum matrix as well as on the surface of the intermetallic particles. The average intermetallic particle size is approximately 0.18 μm . This is slightly larger than the size of the intermetallic dispersion observed in the non-MA material, i.e. 0.11 μm (10,11). The matrix subgrain sizes are comparable in both the MA and non-MA Al-Fe-Ni materials.

The effects of processing history, MA, and exposure temperature on microstructural stability are shown in Figure 2. By comparison with Figure 1, it is clear that the microstructure of the MA material does coarsen at 550 C and above. Increasing the exposure temperature to 610 C, however, does not yield a significant increase in the coarsening rate of the (FeNi)Al₉. This observation pertains to both b and c-processing. Significantly, at 550 C and 610 C, the non-MA material coarsens at a much faster rate than the MA material. Further, the non-MA material is much more sensitive to exposure temperature than the MA material with respect to coarsening rate.

An additional feature in the MA Al-Fe-Ni microstructure exposed at 550 C and 610 C is the appearance of needle-like particles. These do not appear in the as-extruded material nor in the structure exposed at 450 C(20). A representative microstructure after exposure (i.e. a, b, or c- processing) at 550 C is shown in Figure 3. The needle-like morphology of one of the dispersoid species is clearly evident. Similarly, representative microstructures observed after exposure at 610 C are shown in Figure 4; the corresponding microstructure after the same period of exposure at 450 C is

included in Figure 4. Typically, the (FeNi)Al₉ intermetallic increases in size by a factor of about four after exposure at 610 C, compared to exposure at 450 C. The needle-like dispersion present after exposure above 550 C was not observed after exposure at 450 C. TEM observations show a small change in microstructure when the exposure temperature is increased from 550 C to 610 C, Figure 5.

(b) Hardness and Creep Behavior

Hardness measurements of the unexposed and exposed samples are shown in Figure 6. The hardness values for the MA material and the non-MA material obtained from ref. 20 are shown here for comparison. Even after exposure at 610 C, the MA material is stronger than the non-MA material exposed at 450 C, for the range of exposure times examined. Exposure of the non-MA material at 550 C for 50 hours results in a sharp drop in hardness, compared to exposure at 450 C. The small differences in microstructure after coarsening at 550 C and 610 C (Figure 5) are reflected in the hardness values in Figure 6.

In order to investigate the source and nature of the needle-like particles, the non-MA material was also exposed at temperatures of 550 C and 610 C to determine whether these particles are attributable to the MA process. As shown in Figure 7, the non-MA microstructure contains only coarsened intermetallics in the aluminum matrix. Thus, it is believed that these particles result from coarsening of the preexisting Al oxides and/or carbides at temperatures above 500 C, or from precipitation of other oxides/carbides during high temperature exposure. Possible examples are, Fe₂O₃, NiO, FeC₂O₄, or Fe₃C.

In order to compare creep response of the MA and the non-MA material, some tests were conducted on the MA specimens under the same conditions as for the non-MA specimens. Creep data are shown in Figure 8. There is a significant improvement in creep strength brought about by MA.

The MA material was then subjected to a series of creep tests at differing stresses and temperatures. The corresponding creep curves are shown in Figures 9 and 10 for times up to 10 and 100 hours, respectively. There is no tertiary creep in any of the tests. In no material was a true steady state creep rate achieved. Rather, the specimens failed while in the primary creep mode, or the test was terminated before reaching an eventual steady state condition. At low stresses and/or temperatures, the creep specimens exhibited little or no permanent strain; the creep curves extended as a near-horizontal line.

Macroscopically, all the creep specimens tested fractured with little accompanying plastic deformation. Creep fracture surface morphology is illustrated in Figure 11 for MA material exposed at 550 C for 288 hours. The fracture mode is intergranular with extensive grain boundary cavitation.

VI. DISCUSSION

(a) Microstructural Stability and Hardness

For the (FeNi)Al₉ intermetallic to coarsen, diffusion of the Fe and Ni atoms must occur, either via the subgrain boundaries or through the matrix. The present TEM observations (for example Figures 1 and 4) and those reported previously (20) confirm that MA introduces a fine-scale dispersion of oxides/carbides. These are present in the matrix, at matrix-intermetallic interfaces, and at matrix subgrain boundaries. The oxides/carbides on the subgrain boundaries are expected to inhibit diffusion along such paths by acting as vacancy sinks.

With respect to the oxides/carbides at matrix-intermetallic (FeNi)Al₉ interfaces, it is suggested that these dispersions attract the Fe and Ni atoms by forming iron or nickel-base oxides, carbides or oxalates. In consequence, coarsening of the intermetallic is inhibited. It has been shown (10,11) that in the absence of the fine-scale oxides/carbides in the non-MA material, Ostwald ripening proceeds rapidly at 450°C.

Recrystallization in dispersion-strengthened alloy systems is strongly inhibited via the pinning of subgrain boundaries by dispersoids (19,21). In the hot-extruded MA material, both the FeNiAl_9 intermetallic dispersion (size $\sim 0.18\mu\text{m}$) and the fine-scale oxide/carbide dispersion (size $\sim 30\text{nm}$) act to pin subgrain boundaries. At 450C the oxides/carbides severely restrict coarsening of the intermetallic and the subgrain boundaries remain pinned. Above 500°C, the TEM observations show that general coarsening occurs, with an attendant release of the subgrain boundaries.

Exposure at 450C and 550C results in a drop in hardness, in both the MA and non-MA materials. The magnitude of the drop; however, is much larger in the non-MA material, Figure 6. At 450C, only limited coarsening of the FeNiAl_9 intermetallic is observed in the MA material; this leads to a small drop in hardness ($\sim 10\%$). In the non-MA material significant coarsening and grain growth were observed at 450C (10,11), with an attendant large drop in hardness ($\sim 70\%$). In contrast, exposure of the MA material at 550C results in coarsening of $(\text{FeNi})\text{Al}_9$, and possibly of the oxide/carbide dispersions. This results in a larger drop in hardness than at 450C and is attributed to a combination of coarsening and recrystallization, followed by some grain growth. In the non-MA material gross coarsening, recrystallization and grain growth take place at 550C, with an accompanying catastrophic drop in hardness, Figure 7(c).

By means of x-ray diffraction Singer et al (17) showed that in MA aluminum, a fine dispersion of Al_2O_3 and Al_4C_3 is present. The morphology of the oxide/carbide dispersions observed in the present study (e.g. Figure 4) is similar to that reported by Singer et al (17). It is possible that other oxides and/or carbides can form at the higher exposure temperatures. Thus the needle-like particles present after exposure above 550°C (Figures 3, 4, 5 and 7) may be oxides or carbides of Fe and Ni. It is difficult to distinguish between the oxides and the carbides in the MA material.

(b) Creep Response

From Figure 8, it is seen that the creep resistance of the MA material is several orders of magnitude higher than that of the non-MA material. In contrast, the yield strengths of the two materials are similar in magnitude above 350°C (20). It has been suggested that the deformation mechanism(s) operative in creep is independent of the nature of the dispersion (10). The present creep observations do not support this suggestion; the type of dispersion is important in determining creep behavior. Analogous behavior to that illustrated in Figure 8 for the MA versus the non-MA Al-Fe-Ni has been reported in Ag versus Ag+0.1%MgO (22). The creep rate in the dispersion-strengthened alloy was about two orders of magnitude lower than in the single phase Ag.

In primary creep the strain rate decreases rapidly from a large initial value and this is observed in the present study. This change in creep rate reflects the several structural changes occurring in the material, in particular the rearrangement of dislocations with attendant strain hardening. Since the MA Al-Fe-Ni creep specimens fractured with negligible plastic deformation, it can be assumed that the current tests simulate constant stress conditions. Two forms of creep curve are observed in the MA material. Either the material deforms under conditions of transient creep to failure/or termination of the test, or the creep rate becomes zero, Figures 9 and 10.

On a fine microstructural scale, submicron dispersoids enhance creep resistance first by trapping dislocation clusters in cell walls thereby effectively impeding recovery, and secondly by acting as a barrier to diffusion mechanisms. Under these circumstances steady state creep conditions should ultimately be established (23). Not all creep data reported on the basis of minimum creep rate actually correspond to true steady state conditions. This will only pertain if either specimen failure, or a change in specimen geometry, do not intervene.

(c) Processing

No significant differences in microstructure can be observed as a function of processing regime, i.e. a, b, or c - processing. It is clear, however, that differences in strength exist both in the as-extruded condition and following elevated temperature exposure, as a function of processing, Figure 6. Maximum hardness in the hot extruded condition is achieved via b - processing. The resulting material is, however, least resistant to coarsening, especially above 550°C. The most stable material is established by c - processing and is attributed to the combination of a high degassing temperature and a low extrusion temperature. This should minimize microporosity, minimize coarsening during extrusion, and result in a uniform microstructure.

VII. FUTURE STUDIES - Al-Fe-Ce

The ambient and elevated temperature tensile response and microstructural stability of a powder processed Al-Fe-Ce alloy shall be evaluated. The Al-Fe-Ce powder supplied by Alcoa will be mechanically alloyed (MA) and hot extruded to full density by Novamet. Microstructural stability for the MA Al-Fe-Ce alloy with regard to creep behavior shall be evaluated. Based upon the Al-Fe-Ni results, there will be a significant improvement in creep resistance and microstructural stability as compared to the Al-Fe-Ce alloy without MA. These improvements are to be attributed to the presence of fine scale oxides / carbides distributed uniformly throughout the structure and which are introduced during MA process. The fine scale dispersion inhibits coarsening, recovery and recrystallization. The MA alloy should exhibit excellent strength with very limited ductility. Differential scanning calorimetry and transmission microscopy on the non-MA Al-Fe-Ce powders and extrusions shall characterize the thermal response of powders and extrusions e.g. precipitate and dispersoid coarsening response. The combination of mechanical alloying of prealloyed powders provides for excellent microstructural stability with regard to intermetallic coarsening and matrix grain growth. It is anticipated that superior elevated creep response shall

is achieved from the dual rapid solidification / mechanical alloying approach.

III. CONCLUSIONS

- (1) MA of Al-Fe-Ni powder containing 0.19 volume fraction of FeNiAl₉ intermetallic dispersoid (size ~0.18μm) introduces a submicron dispersion (size ~30nm) of oxides/carbides; these are uniformly distributed throughout the aluminum matrix, at matrix-intermetallic interfaces, and on subgrain boundaries.
- (2) MA results in a significant improvement in creep response relative to the non-MA material. MA enhances microstructural stability compared to the non-MA material. Temperatures above 500C are needed to induce coarsening.
- (3) Improvements in microstructural stability are attributed to the presence of the fine-scale oxides/carbides which inhibit coarsening of the FeNiAl₉ intermetallic and grain growth. The fine-scale oxide/carbide dispersoids enhance creep response by impeding diffusion and restricting dislocation motion/rearrangement.
- (4) Processing mode (i.e. degassing/extrusion temperature) does not have a strong influence on microstructural development. Strength, however, monitored in terms of hardness, shows a sensitivity to processing conditions. A combination of a high degassing temperature and low extrusion temperature yields the most stable material.

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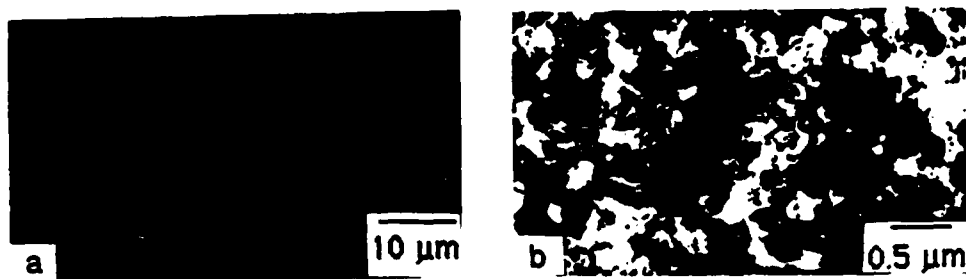


Figure 1: As-extruded microstructure. (a) optical, (b) TEM.

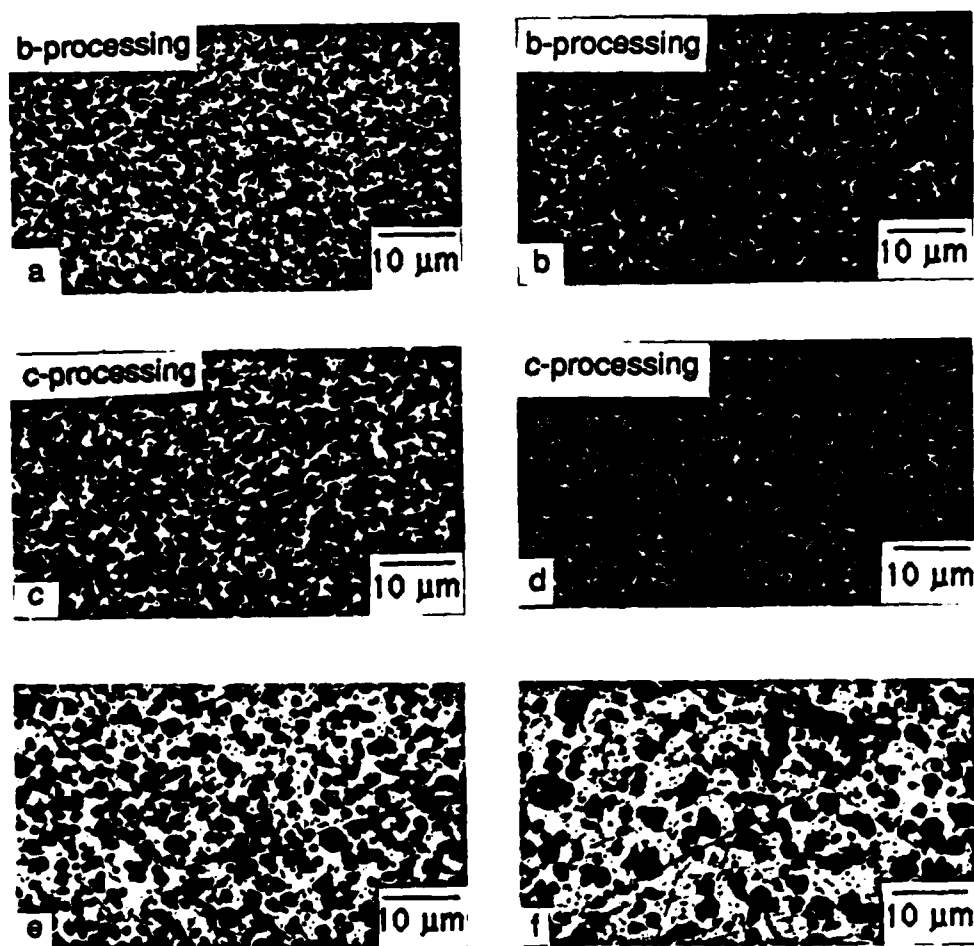


Figure 2: (a) MA, 550°C/50 hr. (b) MA, 610°C/50 hr. (c) MA, 550°C/50 hr. (d) MA, 610°C/50 hr. (e) Non-MA, 550°C/50 hr. (f) Non-MA, 610°C/50 hr.

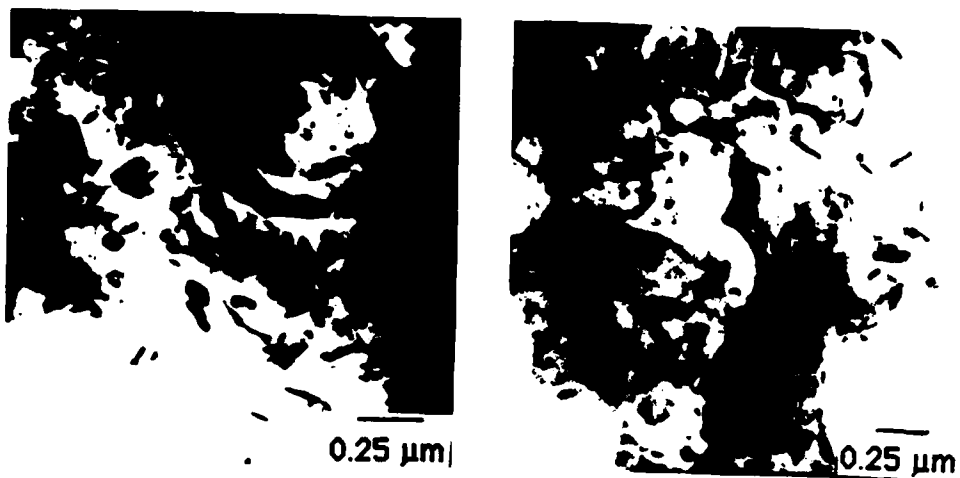


Figure 3: TEM. 550°C/288 hours; MA/a-Processing.

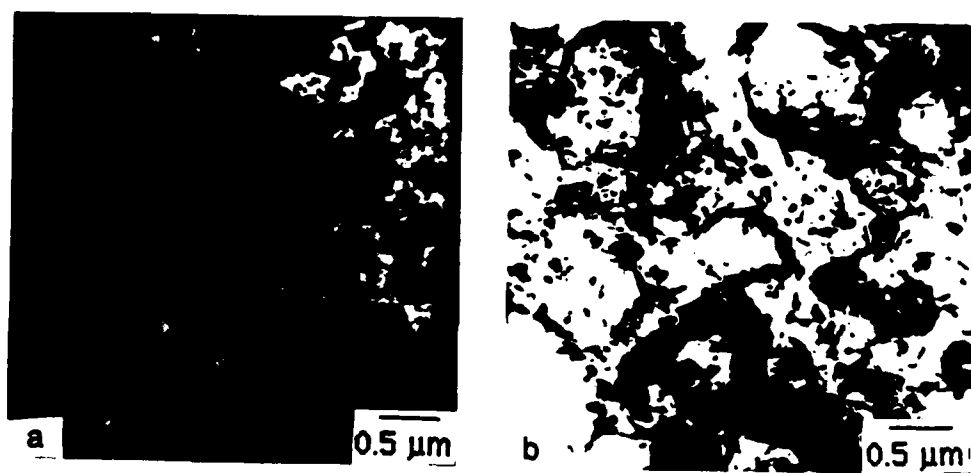


Figure 4: TEM/MA/c-Processing. (a) 450°C/50 hr, (b) 610°C/50 hr.

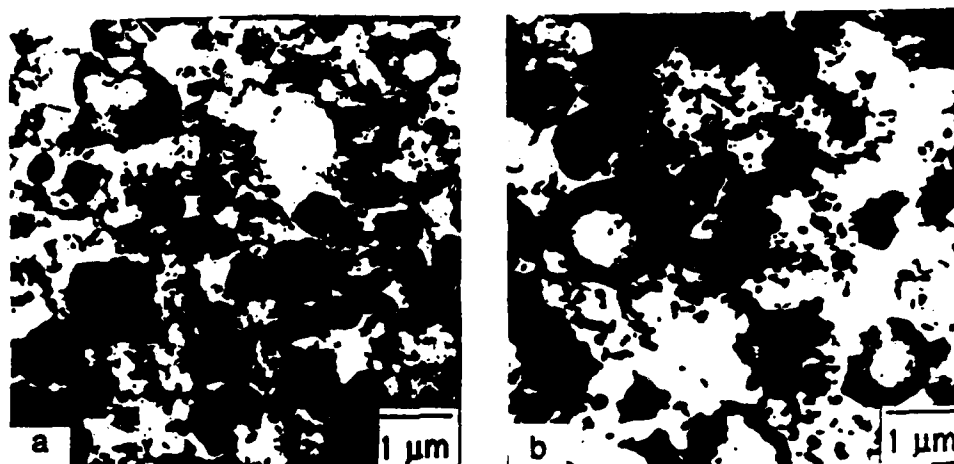


Figure 5: TEM/MA/b-Processing. (a) 550°C/50 hrs, (b) 610°C/50 hrs.

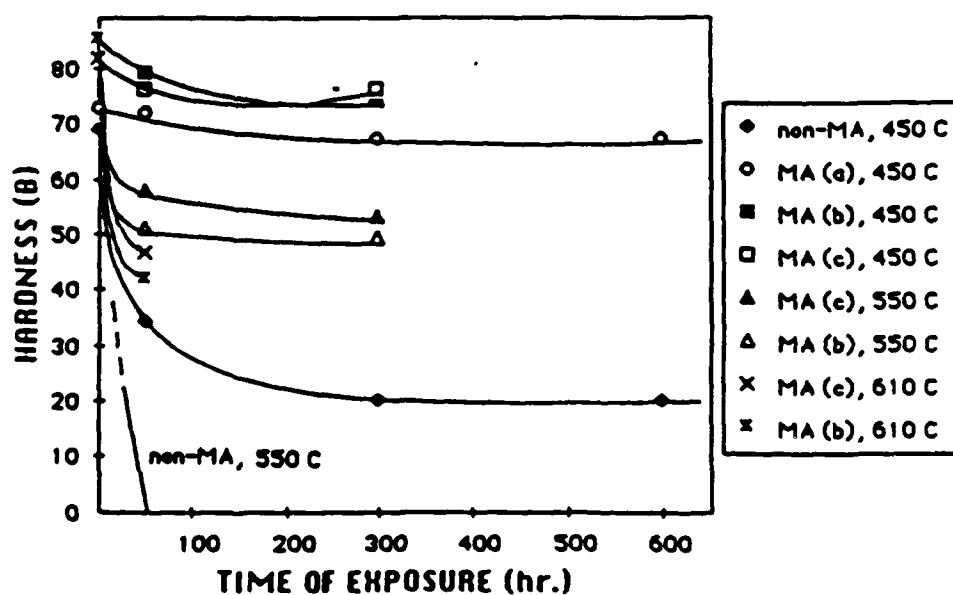


Figure 6: Ambient temperature hardness following elevated temperature exposure. (a), (b) and (c) refer to processing mode.

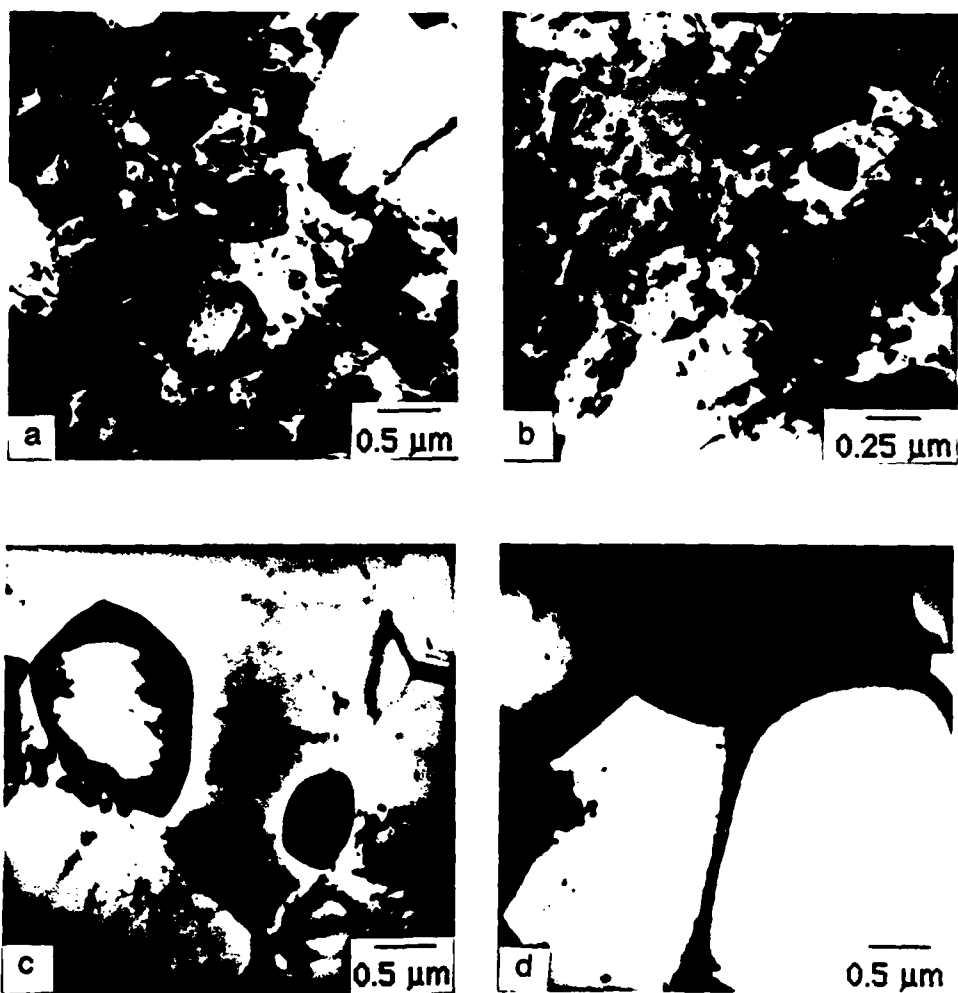


Figure 7: TEM/b-Processing. (a) MA/550°C/50 hrs. (b) MA/550°C/288 hrs. (c) Non-MA/550°C/50 hrs. (d) Non-MA/550°C/50 hrs.

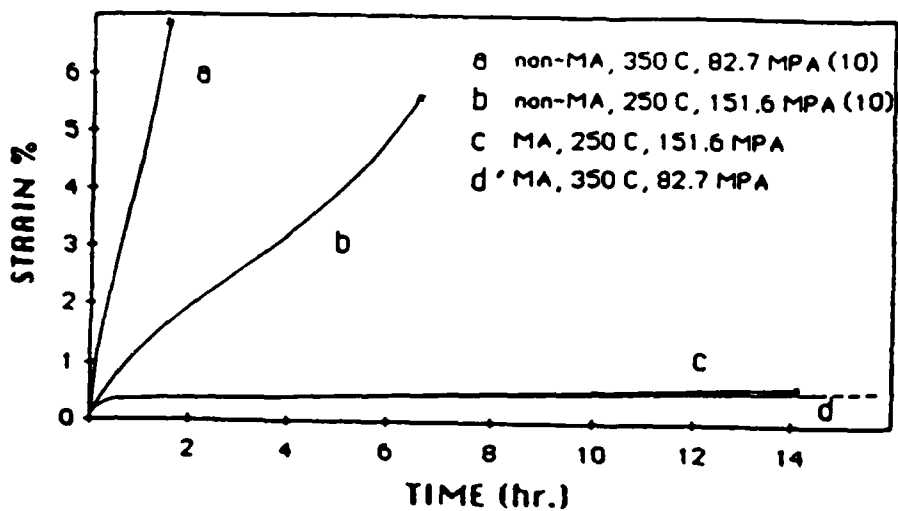


Figure 8: Comparison of creep response of MA and non-MA material.

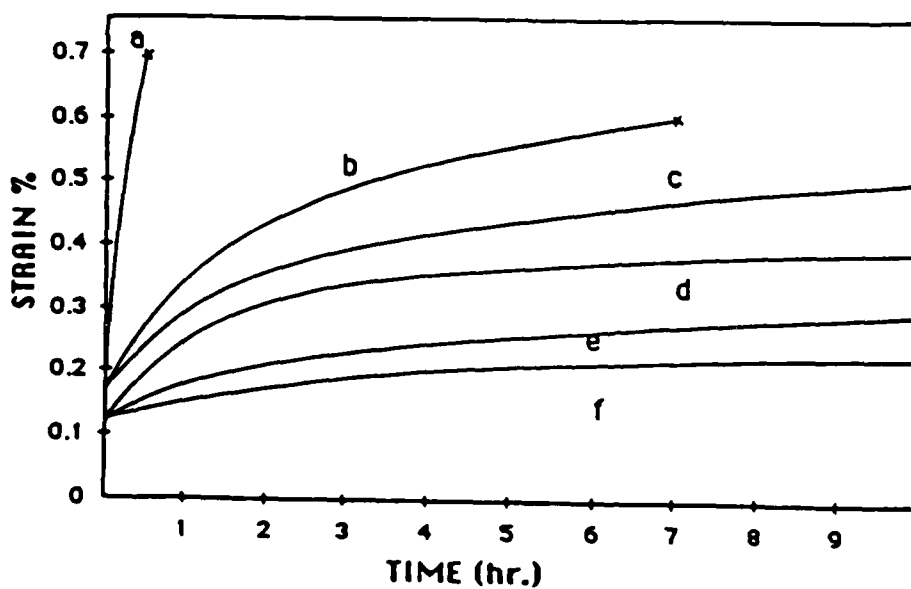


Figure 9: Creep curves of MA material/a-Processing/as-extruded.

- (a) 400°C/90MPa (b) 350°C/103MPa
(c) 350°C/97MPa (d) 350°C/90MPa
(e) 350°C/79MPa (f) 350°C/76MPa

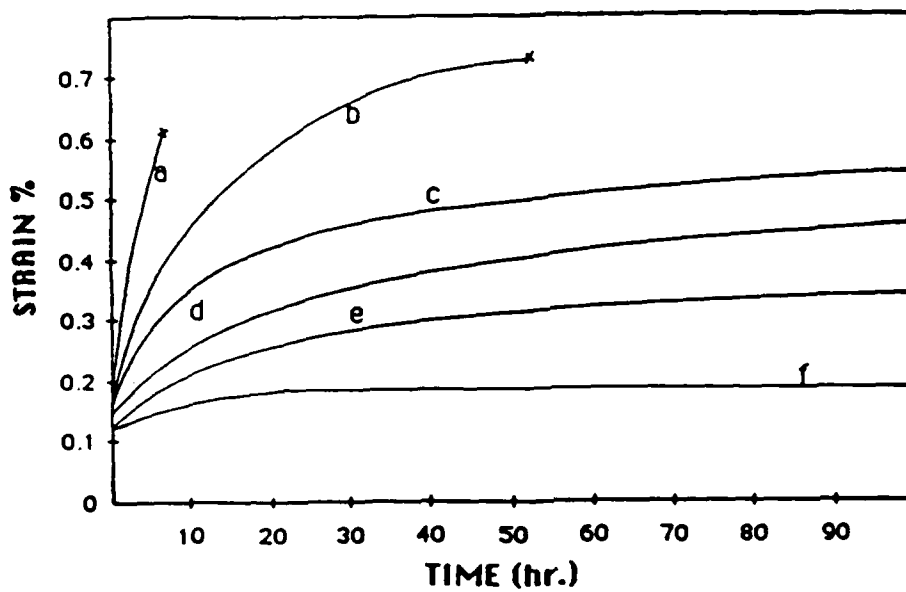


Figure 10: Creep curves of MA material/a-Processing/as-extruded.

(a) 350°C/103MPa (b) 350°C/97MPa
 (c) 350°C/90MPa (d) 350°C/79MPa
 (e) 350°C/76MPa (f) 250°C/103MPa

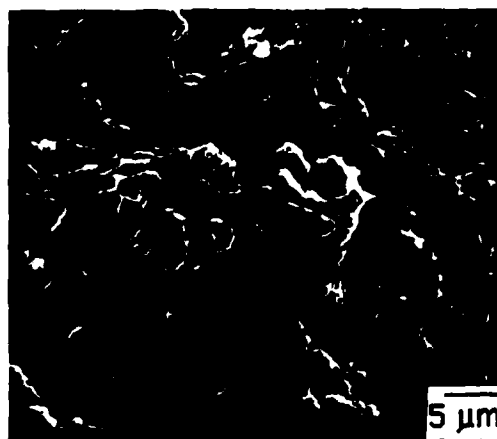


Figure 11: Creep fracture surface morphology. SEM/MA/a-Processing
 Exposure: 550°C/288 hours. Creep: 350°C/76 MPa/failure
 in 7 minutes.

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COUPLING ACTIVITIES

(a) Presentations

"Powder Metallurgy Processing of Dispersion Strengthened Light Metal Alloys", Reynolds Metals Company, Richmond, VA., January 1985.

"Modern Powder Metallurgy Science and Technology", Virginia Section, AIME, Charlottesville, VA., January 1985.

"Rapidly Solidified Materials - Current Assessment and Future Directions", AIME Annual Meeting, New York City, N.Y., February 1985.

"Rapid Solidification Science and Technology", Ford Motor Company, Dearborn, MI, April 1985.

"Aluminum Powder Metallurgy", General Electric R&D Center, Schenectady, NY, May 1985.

"Rapidly Solidified Powder Processes - Models and Mechanisms for Atomization and Consolidation", Northeastern Regional Meeting, The Metallurgical Society of AIME, Morristown, NJ, May 1985.

"The Particle Metallurgy - Rapid Solidification Interface: Atomization Models and Mechanisms", Nicholas J. Grant Symposium, Massachusetts Institute of Technology, Cambridge, MA., June 1985.

(b) Technical Contacts with Other Laboratories

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